
रोगनो, वार्निशों और संबंध उत्पादों के नमूने लेने
और परिक्षण की पद्धतियाँ

भाग 8 वर्णकों और अन्य ठोसों के परीक्षण

अनुभाग 5 सीसा प्रतिबंध परीक्षण

(चौथा पुनरीक्षण)

**Methods of Sampling and
Test for Paints Varnishes and
Related Products**

Part 8 Tests for Pigments and other Solids

Section 5 Lead restriction test

(*Fourth Revision*)

ICS 87.040

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FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Paints, Varnishes and Related Products Sectional Committee had been approved by the Chemical Division Council.

This standard (Part 8/Sec 5) is one of a series dealing with methods of sampling and test for paints, varnishes, and related products. The other sections of this Indian Standard (Part 8) are:

Sec 1 Residue on sieve.

Sec 2 Pigments and non-volatile matter.

Sec 3 Ash content.

Sec 4 Phthalic anhydride.

Sec 6 Volume solids.

This standard prescribed the test methods for determining lead in paints, varnishes, and related products. This standard supersedes clauses 28 and 29 of IS 101 : 1964 Method of test for ready mixed paints and enamels (second revision).

In the third revision three analytical test methods electrolysis, molybdate and sulphide were incorporated for determination of lead in lead restricted paints.

Since, analytical test methods electrolysis, molybdate, and sulphide are not applicable to determine trace quantity of lead in paints, varnishes, and related products. Hence, in this revision, the additional instrumentation test methods Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) and Atomic Absorption Spectroscopy (AAS) methods have been incorporated. Further, the test method for freedom from lead has been removed as it has no significance nowadays.

The Committee responsible for formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, If the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

Indian Standard

METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

PART 8 TESTS FOR PIGMENTS AND OTHER SOLIDS

Section 5 Lead restriction test

(*Fourth Revision*)

1 SCOPE

This standard IS 101 (Part 8/Sec 5) prescribes methods of test for lead restriction in paints and allied products. For lead restriction test any one of the following five methods may be used:

- a) Electrolysis method;
- b) Molybdate method;
- c) Sulphide method;
- d) Atomic Absorption Spectroscopy method (AAS); and
- e) Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES).

NOTES

1 For estimation of lead content of samples ranging below 1 000 ppm the atomic absorption spectroscopy or ICP-OES method should be used exclusively. First three methods are not applicable for determining trace quantities of lead.

2 Electrolysis Method shall be used as a referee method in case of any dispute, if the lead content more than 1 000 ppm and ICP-OES method shall be used as a referee method in case of any dispute if the lead content less than equal to 1 000 ppm.

2 REFERENCE

<i>IS No.</i>	<i>Title</i>
101 (Part 8/Sec 2) : 1990	Methods of sampling and test for paints, varnishes, and related products: Part 8 Tests for pigments and other solids, Section 2 Pigments and non-volatile matter (<i>third revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
264 : 2005	Nitric acid — Specification (<i>third revision</i>)
265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)

3 LEAD RESTRICTION

The paints, varnishes and related products having a lead content below specified limit.

3.1 Electrolysis Method

3.1.1 Procedure

Transfer about 5 g of well mixed paint to a tared evaporating dish and dry at 105 °C to constant mass. Place the exact mass of the dried sample in a muffle furnace and ash it for 20 min, at 315 °C, 40 min at 425 °C and 1 h at 540 °C. Cool in a desiccator. Extract the ash in a 250 ml beaker with 30 ml concentrate nitric acid and 80 ml water and heat to boil. Filter into a 400 ml beaker using fine texture paper to prevent manganese dioxide from passing into the filtrate. Wash with water.

Dilute the filtrate to approximately 300 ml, add 20 ml of 20 percent solution of ammonium nitrate and 10 ml of 0.1 percent copper sulphate solution.

Heat nearly to boiling and electrolyze using platinum gauze anode that has been weighed previously.

Electrolyze for 15 min each at 1A, 2A and then at 3A current. Rinse the electrode three times in water with the current still on. Then remove the anodes, rinse in alcohol, dry for 15 min in an oven at 105 ± 5 °C. Cool and weigh.

NOTE — The electrodes should be cleaned after each determination. This is best done by placing them in nitric acid solution (1 : 4) that contains a few milliliters of concentrated hydrogen peroxide (H₂O₂, 30 percent), rinsed with water and dried for next use.

3.1.2 Calculation

$$\text{Lead, percent by mass on non-volatile matter} = \frac{M \times 0.86623 \times 100}{W}$$

where

M = mass in g, of lead oxide PbO₂; and

W = mass of non-volatile matter taken.

3.2 Molybdate Method

3.2.1 Outline of the Method

Paint is digested with concentrated sulphuric acid and nitric acid in order to convert lead to lead sulphate followed by extraction with ammonium acetate. Finally, lead is precipitated as lead molybdate and weighed as lead molybdate.

3.2.2 Procedure

Transfer about 5 g of well mixed paint to a 400 ml beaker and dry at 105 °C. Weigh accurately, add 20 ml of concentrated nitric acid and 15 ml of concentrated sulphuric acid and digest in order to remove all organic material and change lead to lead sulphate. Remove traces of nitric acid by repeated fuming with sulphuric acid. Cool, add 50 ml of water, heat to dissolve the salts and add 50 ml of ethyl alcohol and set aside for several hours. Filter through a paper pulp pad and wash with dilute sulphuric acid (1 : 20 v/v) containing 10 percent of ethyl alcohol. Dissolve the lead sulphate in three 10-ml portions of hot 20 percent ammonium acetate solution, followed by several washings with hot water. Treat the combined filtrate and washings with 2 ml of glacial acetic acid, heat to boil, add 10 ml of 5 percent aqueous ammonium paramolybdate solution and boil for a few minutes until the lead molybdate precipitate has coagulated.

Filter through a porcelain filtering crucible and wash with ammoniacal 2 percent ammonium nitrate solution. Heat to dull redness at 600-650 °C to constant mass.

3.2.3 Calculation

Lead content, Percent by mass on non-volatile matter

$$= \frac{0.56436 \times W \times 100}{S}$$

where

W = mass of lead molybdate; and

S = mass of dry sample taken for test.

3.3 Sulphide Method

3.3.1 Outline of the Method

Determination of lead in lead restricted paints is carried out by precipitating the lead as sulphide from the separated pigment, which is finally oxidized to lead sulphate.

3.3.2 Procedure

Shake about one gram of the ground pigment obtained after treatment of paints as prescribed in IS 101 (Part 8/Sec 2), accurately weighed, continuously for one hour at room temperature with 1 000 times its weight of an aqueous solution of hydrochloric acid containing 0.25 percent by mass of hydrogen chloride. Allow the mixture to stand for one hour and then filter. Precipitate the lead salt contained in the clear filtrate

as lead sulphide, filter, heat the lead sulphide in air to convert it into lead sulphate, weigh, calculate as lead monoxide (PbO) and express the result as percentage on the dry weight of the material taken for test.

3.3.3 Calculation

$$\text{Lead (as PbO)} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g, of the precipitate; and

M = mass in g, of the sample taken for the test.

3.4 Atomic Absorption Spectroscopy

3.4.1 General Description

This method covers the determination of lead content in pigmented coating, it is also applicable for varnish and lacquer. This method is not applicable for determination of lead in sample containing antimony pigment as low recovery is obtained.

3.4.2 Outline of the Method

The liquid coating or dried film is prepared by dry ashing. The content of lead of an acid extract of ash is determined by atomic absorption spectrophotometer.

3.4.3 Apparatus

3.4.3.1 Atomic absorption spectrophotometer

3.4.3.2 Muffle furnace capable of maintaining temp 500 ± 10 °C

3.4.3.3 Hot plate capable of maintaining temp range 70 to 250 °C

3.4.3.4 Glass beaker 150 ml, 250 ml capacity

3.4.3.5 Porcelain crucible 50 ml or porcelain dish 100 ml capacity

3.4.3.6 Pipette 1, 2, 5, 10 ml capacity.

3.4.3.7 Dropping bottle

3.4.4 Reagent

3.4.4.1 Nitric acid (specific gravity 1.42)

3.4.4.2 Ammonium acetate $NH_4C_2H_3O_2$ (50 percent wt/vol)

3.4.4.3 Lead standard stock solution

3.4.5 Procedure

Mix the liquid coating, mix it until it is homogeneous. Prepare two replicate specimens by weighing by difference from dropping bottle approximately 2 to 3 gm mixed liquid paint. Place the crucible containing liquid coating on hot plate and slowly increase the temp till

it is dried. Gradually increase the temperature of hot plate till the material chars. After charring completely, it is placed in a preheated furnace and ash at 475 to 500 °C. Care should be taken so that the temperature does not exceed 500 °C otherwise there may be a loss by volatilization. When the ashing appears to be completely removed the crucible from the muffle furnace allow to cool to room temperature. Break up the ash in to fine particles with glass rod add 10 ml of diluted nitric acid (1 + 1) taking care to avoid spattering in case the ash reacts vigorously. Heat till the volume reduces to 2 to 3 ml, add additional 10 ml of dil HNO₃ (1 + 1) continue heating.

Filter the solution through filter paper into 50 ml volumetric flask. Wash the container three times with 2.5 ml of hot ammonium acetate solution each time transferring the washing to filter paper. Wash the filter paper several times with water adjust the volume to 50 ml with water and mix.

Aspirate the test solution and determine the absorbance in the same manner in which the instrument was calibrated.

If absorbance is above the range covered by calibration curve dilute an aliquot of sample to a suitable volume.

3.4.6 Calculation

Lead as Pb in ppm

Concentration X dil/wt of the sample.

Lead in percentage multiply the above with 100/10⁶

3.5 Inductively Coupled Plasma-Optical emission Spectrometry

3.5.1 General Description

3.5.1.1 This standard cover procedure for quantitative extraction of lead in dried paint samples (from wet paint in a can or paint powder scraped from a substrate) using hot plate or microwave digestion techniques or both. The method also covers the lead analysis from digested paint samples or extracts using inductively coupled plasma optical (or atomic) emission spectrometry (ICP-OES or ICP-AES).

3.5.1.2 This standard test covers all the products classified as architectural or decorative paints. Accordingly, there will be some differences in sample preparation.

3.5.2 Outline of the Method

Lead in wet paint (in a can) or dried paint sample (film, chips, powder, etc.) is acid extracted (solubilized) by digestion with nitric acid and hydrogen peroxide aided by heating or nitric acid facilitated by microwave energy. The lead content of the digested sample is then analysed using ICP-OES.

3.5.3 Interferences

3.5.3.1 Interferences for ICP-OES can be manufacturer and model specific. The following are general guidelines:

3.5.3.1.1 Some unique interferences may be encountered in the test. These interferences can be minimized by proper wavelength selection, interelement correction factors, and background correction.

3.5.3.1.2 One other Pb line should be used to ensure spectral interferences are not occurring during analysis.

3.5.4 Apparatus

3.5.4.1 Electric hot plate capable of reaching at least 140 °C surface temperature and at least 100 °C when measured by dipping a thermometer inside the digesting solution.

3.5.4.2 Microwave extraction apparatus consisting of a microwave digestion system with power output regulation and fitted with a temperature control system capable of measuring temperature within ± 2 °C. The microwave cavity must be chemical resistant and equipped with exhaust ventilation for acid vapor protection of the unit. All electronics shall be protected against corrosion to ensure safe operation. All safety guidelines of the manufacturer shall be followed.

NOTE — Domestic microwave systems used in household kitchens shall not be used.

3.5.4.3 Sample vessels (closed) of at least 50 ml capacity for microwave digestions capable of withstanding a temperature of at least 180 °C and internal pressure of at least 3 000 kPa. The vessels must be chemical resistant and microwave transparent.

3.5.4.4 Glass beakers, 125-ml or 50-ml with watch glass covers.

3.5.4.5 Volumetric flasks conforming to IS 915, 100 ml and 200 ml.

3.5.4.6 Volumetric pipets or piston-operated pipets conforming to IS 17094 (Part 2), volume as needed.

3.5.4.7 Analytical balance, accurate to 0.1 mg

3.5.5 Reagents

3.5.5.1 *Concentrated nitric acid*, analytical reagent grade (see IS 264) or spectrographic grade (16.0 M HNO₃).

3.5.5.2 *Concentrated hydrochloric acid*, analytical reagent grade (12.3 M HCl) (see IS 265).

3.5.5.3 *Nitric acid*, 10 percent (w/v). Add 100 ml concentrated HNO₃ to 500 ml distilled water and dilute to 1 L.

3.5.5.4 *Hydrogen peroxide*, 30 percent H_2O_2 (w/w); analytical reagent grade.

3.5.5.5 *Water*, conforming to IS 1070

3.5.5.6 *Extraction solution*, mix 500 ml water conforming to IS 1070, 60 ml concentrated HNO_3 and 180 ml concentrated HCl in 1 L volumetric flask and mix well. Allow to cool to room temperature and dilute to 1 L with water.

NOTE — Nitric and hydrochloric acid mists and vapours are toxic. Prepare in a well-ventilated fume hood.

3.5.5.7 *Calibration stock solution*, 100 $\mu\text{g}/\text{ml}$ of lead in dilute nitric acid or equivalent (such as a multielement stock containing lead).

3.5.6 *Sample Preparation*

3.5.6.1 *Sample from dried paint on a substrate*

Carefully scrape some portion of the paint from the substrate. Ensure that the sample doesn't contain excess substrate. It may be necessary to add a few drops of solvent, such as methylene chloride, to soften the paint and aid in its removal from the substrate. If used, such solvent must be evaporated fully prior to analysis. The scraped paint should be finely divided to help in digesting. If multiple samples are taken from substrate, then homogenize them using pestle and mortar and take out required quantity for testing.

3.5.6.2 *Wet paint and colorant*

Apply a thin coating (or colorant) to a glass slide, and dry completely prior to testing by heating in an oven at nominally 105 °C (105 °C + 2 °C) until the weight is stable for at least two successive readings, the readings should be separated by 30 min of heating in the oven. Scrape out the paint film and ensure that it is finely divided. General cold grinding processes like dry ice assisted grinding or liquid nitrogen grinding will help in finely dividing the sample.

3.5.6.3 *Two pack (or more) products*

Mix the multiple components of the paint as recommended in product data sheet and spread it as a thin layer (or as a draw down) on glass plate or a silicone release paper. Cure the film in accordance with manufacturer recommendation and pick up the sample as described in **3.5.6.1**.

3.5.6.4 *Thinners or diluents*

These samples shall be taken as it is for analysis.

3.5.6.5 *Hot plate digestion*

3.5.6.5.1 Weigh 0.25 — 0.5 g of the homogenized sample to the nearest 0.1 mg and place it into a clean, labelled 125-ml or 50-ml beaker.

3.5.6.5.2 Add 3 ml concentrated HNO_3 and 1 ml 30 percent H_2O_2 , and cover with a watch glass. Place the beaker with the contents on a hot plate preheated to 85-100 °C. This step is necessary to avoid spattering of content. Increase the hot plate temperature to 140 °C and continue the heating until most of the acid has evaporated leaving less than 1 ml in the bottom of the beaker. In this process, do not allow the content to boil or splash. Remove the beaker from the hotplate and allow it to cool to room temperature.

NOTE — Exercise caution, solution may quickly dry up.

3.5.6.5.3 Repeat step **3.5.6.5.2** two more times using 3 ml concentrated HNO_3 and 1 ml 30 percent H_2O_2 . Heat (surface temperature approximately 140 °C) until the sample is nearly dry. Evaporate to near dryness.

3.5.6.5.4 Rinse the watch glass and beaker walls with 3 to 5 ml 10 percent HNO_3 and allow the solution to evaporate gently to dryness (surface temperature approximately 140 °C). Allow to cool to near room temperature. Add 2 ml concentrated HNO_3 to the residue, swirl to dissolve soluble species. Rinse the beaker walls and bottom of the watch glass with water and quantitatively transfer to a 100 ml volumetric flask. Dilute to volume with water.

3.5.6.5.5 Remove any particulate in the digestate by filtration, centrifugation or allowing the sample to settle prior to instrumental measurement. The diluted digestate solution contains approximately 2 percent (v/v) nitric acid. Calibration standards used for instrumental measurement should be made with this level of nitric acid.

NOTE — Nitric acid mists and vapours are toxic. Perform the following operations in a fume hood or an appropriately ventilated area.

3.5.6.6 *Microwave digestion*

3.5.6.6.1 Weigh 0.25 – 0.5 g of the homogenized sample to the nearest 0.1 mg and transfer into the clean liner of a labelled microwave digestion vessel.

3.5.6.6.2 Carefully add 10 ml of concentrated nitric acid or extraction solution (**3.5.5.6**) to the inside liner of the digestion vessel containing the sample or blank. Seal the vessels in accordance with manufacturer's instructions. Gently swirl the contents and allow them to stand undisturbed for 20 min.

3.5.6.6.3 Load the vessels into the microwave oven in accordance with manufacturer's instructions. Vessels containing samples shall be evenly and symmetrically placed in the microwave oven. Program the microwave digestion system to reach at least 180 °C (65 °C) in less than 10 min, and then hold at this temperature for at least 15 min.

3.5.6.6.4 Remove the vessels from the microwave oven, place them in a fume hood and allow the solutions to cool to room temperature. Carefully detach the vent tubing and shake the vessels to release any excess gas pressure.

3.5.6.6.5 Carefully open the vessels and transfer the contents into 50 ml volumetric flasks. Carefully rinse the vessel with water (3-4 times) and bring to volume in the 50 ml. Seal each flask with a stopper and mix thoroughly.

3.5.6.6.6 Carefully transfer entire contents of volumetric flask into a polypropylene 50 ml centrifuge tube or a Tarson tube. Centrifuge the sample solution at 3 000 rpm for 10 min. The sample is now ready for aspirating into spectrophotometer. If necessary, filter the solution through 0.45µ nylon syringe filter.

3.5.7 Procedure

3.5.7.1 Set up the spectrometer for the analysis of lead at a primary lead emission line (such as 220.353) in accordance with the instructions given by the manufacturer. Be sure to allow at least a 30 min warm-up of the system prior to starting the calibration and analysis.

3.5.7.2 Preparation of calibration standards.

3.5.7.2.1 Add 20 ml of concentrated HNO₃ into a 1 l volumetric flask containing 500 ml water and dilute to the mark. This solution is used as diluent in subsequent steps, and it corresponds to 2 percent HNO₃ (v/v).

3.5.7.2.2 Add 1.25 ml of 100 ppm lead standard solution into a 25 ml volumetric flask and dilute with 2 percent HNO₃ to the mark. This solution corresponds to 5 ppm standard lead solution, and it shall be used as stock solution to prepare series of standards.

3.5.7.2.3 Prepare 0.01, 0.02, 0.05, 0.1, 0.4, 0.8 and 1.0 ppm standard lead solutions by diluting 0.05, 0.1, 0.25, 0.5, 2.0, 4.0 and 5.0 ml of stock solution (see 3.5.7.2.2) respectively in 25 ml volumetric flasks with 2 percent HNO₃. 2 percent HNO₃ solution shall be used as initial calibration blank (ICB). Prepare a QC check standard by diluting 1.75 ml of stock solution to 25 ml in a separate volumetric flask.

3.5.7.3 Set up the following parameters in the instrument.

RF power	1150 W
Nebulizer gas flow	0.5 l/min
Coolant gas flow	12 l/min
Auxiliary gas flow	0.5 l/min
RPM	50
Mode	Axial
Detector wavelength	220.353 nm

3.5.7.4 Aspirate the calibration standard solutions prepared in 3.5.7.2 and monitor the results. Coefficient of regression for calibration shall be ≥ 0.995 .

3.5.7.5 Aspirate the ICB sample followed by QC check standard and again ICB. Failure to meet the requirements of ICB and QC check standard require complete recalibration of the instrument.

3.5.7.6 Run ICB followed by digestate or extract of the sample. QC check standard shall be used after analysing every 10 samples.

3.5.8 Calculation

3.5.8.1 Calculation of lead concentration in sample digestate/extract

Calculate the lead concentration in the sample digest or extract after instrumental analysis as follows:

$$\text{Measured lead in sample solution, ppm } (\mu\text{g/ml}), A = (Ai) (D)$$

where

Ai = instrumentally measured lead concentration, ppm (µg/ml); and

D = dilution factor, ml/ml, required during instrumental analysis to produce a measured lead level within the calibration curve.

3.5.8.2 Calculation of lead concentration in paint samples

Calculate the lead concentration in the original paint sample after instrumental analysis as follows:

$$\text{Lead mass per unit mass of sample, ppm } (\mu\text{g/ml}) = [(A) (B)]/(C)$$

where

A = measured lead content in sample, digestate or extract from 3.5.8.1;

B = final dilution volume, ml; and

C = sample mass, g.

NOTE

1 Interferences are common in determination of lead from coloured paint samples. Paints containing colour pigments derived from metals (for example aluminium paste, copper paste, etc) or organometallic compounds such as copper phthalocyanines strongly interfere when lead is estimated at 220.353. In such cases, appropriate secondary wavelengths must be selected as explained in 3.5.3.1 (for example, 182 nm). In any case, it is necessary to check interference of other metals at wavelength of choice.

2 This method can be used for estimation of lead in paint raw materials. In such case, sample weight can be in the range of 0.1-0.25 g. Extreme care and caution need to be exercised while extracting lead from raw material in microwave digestion process.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Paints, Varnishes and Related Products Sectional Committee, CHD 20

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Amendments Issued Since Publication

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